

## Claims

- [c1] A method for making an ultra-fine, submicron grain titanium or titanium-alloy article comprising the steps of: providing a coarse grain titanium or titanium-alloy material having a first grain size; cryogenically milling the coarse grain titanium or titanium-alloy material into an ultra-fine, submicron grain material having a second grain size less than the first grain size; degassing the ultra-fine, submicron grain titanium or titanium-alloy material; densifying the ultra-fine, submicron grain material to form a densified ultra-fine grain material; and forming the article from said densified ultra-fine, submicron grain titanium or titanium-alloy material.
- [c2] The method of claim 1, wherein forming comprises without subsequent thermal processing.
- [c3] The method of claim 1, further comprising thermal processing after forming.
- [c4] The method of claim 1, wherein the ultra-fine, submicron second grain size material is in the nanocrystalline

range.

- [c5] The method of claim 1, wherein densifying the ultra-fine, submicron grain material to form a densified ultra-fine, submicron grain material comprises hot isostatic pressing the ultra-fine, submicron grain material to form a densified ultra-fine, submicron grain material.
- [c6] The method of claim 1, wherein densifying the ultra-fine, submicron grain material to form a densified ultra-fine, submicron grain material comprises Ceracon-type forge consolidating the ultra-fine, submicron grain material to form a densified ultra-fine, submicron grain material.
- [c7] The method of claim 1, wherein densifying comprises densifying the material in an at least partially nitrogen atmosphere.
- [c8] The method of claim 1, wherein densifying comprises densifying the material in an at least partially argon atmosphere.
- [c9] The method of claim 1, wherein forming comprises extruding.
- [c10] The method of claim 1, an article formed according to the process of claim 1.

- [c11] The method of claim 1, wherein said titanium–alloy material is composed of Ti–6Al–4V.
- [c12] The method of claim 1, wherein said titanium–alloy material is composed of commercially pure titanium.
- [c13] The method of claim 1, wherein said titanium–alloy material is composed of Ti–5Al–2.5Sn.
- [c14] The method of claim 1, wherein said titanium–alloy material is selected from the group consisting of binary titanium compositions, such as  $\beta$ -Ti–Mo and  $\alpha$ -Ti–Al.
- [c15] The method of claim 1 wherein the cryogenically milling comprises cryogenically milling until the grain material is sized to between about 100–500 nanometers.
- [c16] The method of claim 1 wherein the cryogenically milling comprises cryogenically milling until the grain material is sized to between about 100–300 nanometers.
- [c17] The method of claim 1 wherein cryogenically milling is performed in an at least partially nitrogen atmosphere or at least partially argon atmosphere.
- [c18] The method of claim 1, wherein the steps of milling comprises:  
introducing said titanium or titanium–alloy material to a

stirring chamber of a cryogenic milling device;  
contacting said titanium or titanium–alloy material with a milling medium for a pre–determined amount of time sufficient to impart mechanical deformation into said coarse–grained titanium or titanium–alloy material to form an ultra–fine, submicron grain structure on said titanium or titanium–alloy material; and  
removing said ultra–fine, submicron grain titanium or titanium–alloy material from said stirring chamber through an outlet or other method.

[c19] The method of claim 18, wherein the ultra–fine, submicron grain structure has a grain size between approximately 100 and 400 nanometers.

[c20] The method of claim 18 wherein the ultra–fine, submicron grain structure has a grain size between approximately 100 and 300 nanometers.

[c21] The method of claim 18, wherein the step of providing a coarse–grain titanium or titanium–alloy material having a first grain size comprises the step of providing a coarse–grain titanium or titanium–alloy material having a grain size of approximately 0.05 millimeters.

[c22] The method of claim 18, wherein the step of mechanically–forming an article from said ultra–fine, submicron

grain titanium or titanium–alloy material comprises the step of cold–working an article from said ultra–fine, sub–micron grain titanium or titanium–alloy material.

[c23] The method of claim 18, further comprising cooling said titanium or titanium–alloy material to about a liquid hydrogen temperature.

[c24] A method as recited in claim 1 wherein milling comprises milling with a stearic acid additive.

[c25] A method as recited in claim 14 further comprising the steps of:

introducing the ultra–fine, submicron grain titanium or titanium–alloy material within a cavity of a mechanical forming die, said cavity having the general shape of the fastener or other fastening article or device;

cutting said ultra–fine, submicron grain titanium or titanium–alloy material;

removing said cut ultra–fine, submicron grain titanium or titanium–alloy material from said cold–forming die.

[c26] The method of claim 25 further comprising artificially–aging said cut ultra–fine, submicron grain titanium or titanium–alloy material.

[c27] The method of claim 25, wherein the step of introducing an ultra–fine, submicron grain titanium or titanium–alloy

material within a cavity of a cold-forming die comprises the step of introducing an ultra-fine, submicron grain titanium or titanium-alloy material within a cavity of a cold-forming die using a ram.

[c28] The method of claim 25, wherein the step of cutting said ultra-fine, submicron grain titanium or titanium-alloy material comprises the step of cutting said ultra-fine, submicron grain titanium or titanium-alloy material using a shear device.

[c29] The method of claim 25 wherein the ultra-fine, submicron grain structure has a grain size between approximately 100 and 400 nanometers.

[c30] The method of claim 25 wherein the ultra-fine, submicron grain structure has a grain size between approximately 100 and 300 nanometers.

[c31] A method for making an ultra-fine, submicron grain titanium or titanium-alloy article comprising the steps of: providing a coarse grain titanium or titanium-alloy material having a first grain size; cryogenically milling the coarse grain titanium or titanium-alloy material into an ultra-fine grain material less than the first grain size in a supercooled mixing chamber;

degassing the ultra-fine, submicron grain titanium or titanium-alloy material;  
consolidating the ultra-fine, submicron grain material to form a densified ultra-fine grain material; and  
forming the article from said densified ultra-fine, submicron grain titanium or titanium-alloy material.

- [c32] A method as recited in claim 31 wherein the first grain size is about 0.05 millimeters.
- [c33] A method as recited in claim 31 wherein the ultra-fine second grain size is between 100 and 500 nanometers.
- [c34] A method as recited in claim 31 wherein the ultra-fine grain size is between 100 and 300 nanometers.
- [c35] A method as recited in claim 31 wherein cryogenically milling comprises cryogenically milling titanium in a slurry with liquid nitrogen.
- [c36] A method as recited in claim 35 wherein cryogenically milling comprises cryogenically milling titanium in a slurry with liquid nitrogen and argon or helium.
- [c37] A method as recited in claim 31 wherein cryogenically milling comprises cryogenically milling titanium in a slurry with liquid argon or liquid helium.
- [c38] A method as recited in claim 31 wherein consolidating

comprises consolidating using an HIP process.

[c39] A method as recited in claim 31 wherein consolidating comprises consolidating using a Ceracon-type forge.

[c40] A method as recited in claim 31 wherein milling comprises milling with a stearic acid additive.